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REPORT NO. 710/412

# ETCHING OF CARBIDES IN STEEL BY MEANS OF MURAKAMI'S REAGENT

by

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February 17, 1942
WATERTOWN ARSENAL
WATERTOWN, MASS.

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Report No. 710/412
Watertown Arsenal
(Ex. O. 51-A2, 1941)

February 17, 1942

# Etching of Carbides in Steel

# by Means of Murakami's Reagent

## OBJECT

To investigate the effect of this reagent on various carbides in different types of steel in order to better understand its applications and limitations.

## REFERENCES


1. Metals Handbook - American Society for Metals, 1939 Edition.

2. Metallographers' Handbook of Etching - Berglund.

3. Structure of Martensitic Carbon Steels and Changes in Microstructure Which Occur Upon Tempering - H. S. Rawdon and S. Epstein, Scientific Paper of Bureau of Standards No. 452.

4. Use of Sodium Picrate in Revealing Dendritic Segregation in Iron Alloys - A. Sauveur and V. N. Krivobok - Transactions, A.I.M.M.E., Vol. LXX, 1924.

5. Watertown Arsenal Report No. 710/408.


## CONCLUSIONS

1. Carbides can be detected by their darkened appearance after etching in Murakami's reagent, but no differentiation between the various complex carbides can be made. (See Figures 1a -5b, 8a - 10d.)

2. Segregations of carbides can be seen more easily when the steel is etched in Murakami's reagent than when it is etched in 1% nital. (See Figures 5a, 5b, 10a, and 10b.)

3. A light obscuring film is deposited on the free iron carbide present in unalloyed high carbon steels. Then etched with Murakami's reagent this film must be removed by a slight repolish before the blackened condition of the iron carbide is revealed. (See Figures 6a, 6b, 6c, 6d, 7a, 7b, and 7c.)

4. The use of alkaline sodium picrate is not recommended for etching carbides since this reagent attacks constituents and segregations other than carbides. (See Figures 11d - 13e inclusive.)

5. A 10% chromic acid electrolytic etch has proven very successful in revealing the presence of carbides in armor plate of the low carbon variety. The carbides in this same plate are not clearly revealed by the Murakami reagent. (See Figures 11a, and 11b.)

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## INTRODUCTION

Since recent investigations on the correlation of the microstructure and ballistic properties of armor plate have involved the extensive use of Murakami's reagent, this investigation was made in order to determine its merit in etching carbides in various types of steels.

## TEST PROCEDURE

Materials used for this investigation were commercial steels, special vacuum melted alloys, and armor plates. A complete list of the materials examined are shown in Table II. Of the commercial steels, No. 7 was a hot rolled 1/4 inch rod, No. S was commercially spheroidized steel, Nos. 19, 67, and 69 were cast steels. The special alloys were prepared* from Armco iron bases to which the alloying elements were added, melted in vacuum, and annealed at 1600°F for half an hour. The armor plates included a face hardened plate with a very brittle case and five homogeneous plates, two of good quality, one which spalled, one with a low ballistic limit, and one which buttoned and cracked in the ballistic test.

The specimens were polished and etched in 1% nital, then photographed. The nital etch was then removed by careful repolishing with levigated alumina. The specimens were then re-etched, this time in Murakami's reagent, at a temperature just below 100°C for periods noted in Table I. Care was taken to photograph the same spot on the specimen with this etch as had been photographed with the nital etch. In some cases, see Figures ~~20~~, 6d ~~21~~, and 7C ~~24~~, a light and careful retouching on the polishing cloth was necessary to remove a film formed on the surface of free cementite.

Microscopic examinations and photomicrographs were made with a 4 mm. apochromatic objective of numerical aperture 0.95 and with a green light filter.

The formula for Murakami's reagent is 10 grams of potassium ferricyanide and 10 grams of potassium hydroxide, in 100 cc of water, the solution to be freshly made for each etch. All etching was done in a solution held just below the boiling point.

Several samples of armor plate, for which the Murakami reagent proved unsatisfactory, were etched in alkaline sodium picrate and in 10% chromic acid (electrolytic etch) in order to determine the merit of these reagents in etching carbides.

Etching in alkaline sodium picrate consisted of etching in this reagent for five minute intervals, the temperature being kept just below the boiling point. It was found that a 25-minute etching period of five 5-minute cycles produced the best degree of etching for both visual and photographic purposes.

---

*Made by E. L. Reed, Harvard University.

Electrolytic etching in 10% chromic acid was conducted as follows:

| | | |
|---|---|---|
| anode | - | sample to be etched |
| cathode | - | stainless steel strip |
| current | - | 15 amps. 6 volts |
| solution | - | 10% chromic acid |
| time | - | 15-45 seconds |

## RESULTS

1. Etching in a boiling solution of Murakami's reagent generally causes considerable staining of the specimen. A more satisfactory result is achieved when the temperature of the solution is maintained just below 100°C. (Preferably by heating Murakami's reagent in a water bath.)

2. Depending upon the carbon content, the alloying elements, and the microstructure of the steel, the time required to etch a particular steel varies from one second to thirty minutes. These results are summarized in Table I.

a. Steels containing either manganese, molybdenum, vanadium, or tungsten, or plain carbon steels of approximately 0.5% carbon, etch normally in fifteen minutes.

b. High carbon steels are the most resistant to the reagent and require the longest periods of etching.

c. The presence of chromium, (probably as a complex chromium-iron-carbide), causes the steel to etch rapidly, as does a high phosphorous content.

d. Steels containing very fine carbides require longer periods in the hot solution to etch properly than do steels whose carbides are in coarser particles. (See Figures 4 and 18.)

3. As a result of literature search and partially confirmed by laboratory tests, it is believed that alkaline sodium picrate is not a suitable carbide etching reagent for the following reasons:

a. Alkaline sodium picrate does not always blacken cementite of high chromium content.

b. In austenitic and pearlitic manganese steels, alkaline sodium picrate will bring out the distinctive banded appearance of manganese segregation.

c. In silicon steels, an alkaline solution of sodium picrate blackens ferrite containing silicon.

d. Alkaline sodium picrate blackens manganese sulphide inclusions and darkens regions rich in these inclusions and segregated areas higher in manganese than the matrix.

e. Alkaline sodium picrate etches martensite very readily and may thus lead to false conclusions.

4. Difficulty has been experienced in revealing carbide segregations in armor plate compositions containing .18/.23% carbon, after etching in the Murakami reagent. An electrolytic etch in 10% chromic acid has proven satisfactory.

## DISCUSSION

The results of this investigation indicate that Murakami's reagent may be used to reveal carbides of medium and large particle size in most of the commercial steels. (See Figures 1a-5b; 8a-10d.)

It is necessary to etch longer those steels in which the carbide particles are extremely fine. For example, the plain carbon steel shown in Figure 1d was etched twenty minutes, and the iron carbide at the grain boundaries and in the lamellae of the pearlite etched thoroughly in this length of time. On the other hand, the plain carbon steel shown in Figure 5b and which has a much finer structure required thirty minutes of etching to reveal the very fine carbides properly. It is interesting to notice that in these steels repolishing was not necessary to reveal the darkening of the carbides, since on small areas such as the carbides in these steels present, there is no tendency to deposit the obscuring film which occurs in the more massive carbide areas shown in Figures 6d and 7c.

The failure of the troostitic structures of Figure 8b to appear in Figure 8c is probably due to the fact that one second in the solution was not sufficient time for the etch to attack these fine carbides. The extremely short etching time had to be employed since the complex chromium-tungsten-carbides in the eutectic etch so rapidly. In steels such as this a longer interval in a cold solution would probably be a more satisfactory method of etching.

The nital etch does not always reveal the presence of carbides as clearly as the Murakami etch. (See Figures 5a, 5b, 10a, and 10b.)

The long period required to etch unalloyed high carbon steels produces a light colored film on the free iron carbide. When this film is removed by a slight repolishing with levigated alumina, the blackened condition of the free cementite is revealed. (See Figures 6a, b, c, and d for an example of spheroidized steel, carbon 1.27%, and Figures 7a, b, and c for a cast steel containing 1.62% carbon.

Carbides in the medium high carbon Cr-Mo-V homogeneous armor plate and in the carburized case of the carburized Ni-Mo armor plate are clearly revealed by the Murakami etch. (See Figures 9a-10d inclusive.)

When alkaline sodium picrate is used alone as an etching reagent for the presence of carbides, it may often lead to erroneous conclusions, see Figures 11d and e, 12a, b, and c, but when used in conjunction with etching reagents whose specific effects are well understood the use of alkaline sodium picrate may help confirm the conclusions previously arrived at, such as Figures 11b and 11c. Sometimes pronounced banding in steels which is the result of causes other than carbide segregations is revealed by alkaline sodium picrate. (See Figures 12a, b, c, and 13a, and b.) In the case of a relatively high carbon band in the central layers of a Ni-Mo homogeneous armor plate, the sodium picrate attacked constituents other than carbides. (See Figure 11e.) This same area is etched normally with the 10% chromic acid (electrolytic etch). (See Figure 11d.) Furthermore, according to investigations in other laboratories and partly confirmed at Watertown Arsenal, alkaline sodium picrate etches martensite, Figures 13c, and d, and also etches segregations in various types of steels. (See Figure 13e.) Sodium picrate may reveal macrostructure and microstructure.

It has been noted that the carbides in low carbon armor plate are more clearly revealed by electrolytic etching in 10% chromic acid than by the Murakami reagent. (See Figures 11a, and b.)

TABLE I

| Specimen No. | Type of Alloy | Etch in Murakami's Reagent: Microstructure | Time in Minutes | Reference to Figure No.: Nital | Murakami |
|---|---|---|---|---|---|
| 234 | Vacuum melt-Armco iron base 0.48% C | $Fe_3C$ in pearlite | 20 | 1a | 1b |
| 437 | " " " " " 1.06% C | Free & eutectoid $Fe_3C$ | 20 | 1c | 1d |
| 600 | " " " " " 1.00% C&.26% P. | Steadite and eutectoid $Fe_3C$ | 1 | 2a | 2b |
| 414 | " " " " " .52% C, 12.18% Cr. | Chromium carbide segregations & fine chromium carbides. | ½ | 2c | 2d |
| 400 | Vacuum melt-Armco iron base 1.34% C, 6.58% Cr. | Chromium carbide segregations and fine chromium carbides. | 10 | 3a | 3b |
| 97 | Vacuum melt-Armco iron base .45% C, 6.0% Mo. | Molybdenum carbide segregations and fine molybdenum carbides. | 15 | 3c | 3d |
| 257 | Vacuum melt-Armco iron base .49% C, 2.35% V. | Segregated vanadium carbides and fine vanadium carbide. | 15 | 4a | 4b |
| 209 | Vacuum melt-Armco iron base .50% C, 8.48% W. | Segregated tungsten carbides and fine tungsten carbides. | 15 | 4c | 4d |
| 7 | Commercial rolled .795% C steel. | Fine & coarse pearlite. | 30 | 5a | 5b |
| S | " " 1.27% C steel. | Removal of film from spheroidized cementite showing blackened condition of $Fe_3C$. | 30 | 6a | 6b,6c,6d |

(Continued)

TABLE I (Cont'd)

| Specimen No. | Type of Alloy | Etch in Murakami's Reagent: Microstructure | Time in Minutes | Reference to Figure No.: Nital | Murakami |
|---|---|---|---|---|---|
| 19 | Commercial cast 1.62% Carbon steel | Removal of film from cleavage $Fe_3C$ showing blackened condition of $Fe_3C$. $Fe_3C$ in pearlite attached. | 30 | 7a | 7b, 7c |
| 67 | Commercial as cast Hadfield manganese steel, 1% C, 13% Mn. | $Mn_3C$ present in grain boundaries, needles & patches. | 15 | 8a | 8b |
| 69 | Commercial as cast high speed steel, .60% C, 18% W, 4% Cr, and 1% V. | Eutectic structure. | 1/60 | 8c | 8d |
| E-7/8 | Poor quality face hardened armor plate 5% Ni, .2% Mo. | Grain boundary carbides in carburized case. | 10 | 9a | 9b |
| N10-3 | Poor quality homogeneous armor plate 1.1% Cr, .6% Mo, .2% V. | Segregations of grain boundary carbides. | 10 | 9c | 9d |
| P5-3 | " " " | A few segregations of grain boundary carbides. | 10 | 10a | 10b |
| N4-3 | Good quality, homogeneous armor plate 1.1% Cr, .6% Mo, .2% V. | Absence of grain boundary carbides. | 10 | 10c | 10d |
| 1830-D4* | Good quality homogeneous armor plate, 5% Ni, .20% Mo. | Some carbides present in grain boundaries. | - | - | - |
| 5A* | Good quality homogeneous armor plate, 1.1% Cr, .6% Mo, .2% V. | | | | |

*These samples were etched in Murakami's reagent, 10% chromic acid (electrolytic etch) and alkaline sodium picrate. (See Figures 11a, 11b, 11c, 11d, 11e, 12a, 12b, and 12c.)

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Table II

Chemical Analyses

| Specimen Identification No. | C | Mn | P | S | Si | Cr | Mo | V | W | Cu | Ni |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Vacuum Melts | | | | | | | | | | | |
| 234 | .48 | .079 | .005 | .029 | .02 | -- | -- | - | - | -- | -- |
| 437 | 1.06 | .08 | .004 | .003 | .17 | -- | -- | - | - | .048 | -- |
| 600 | 1.00 | .061 | .26 | .004 | .140 | -- | -- | - | - | .048 | -- |
| 414 | .52 | .079 | .005 | .029 | .02 | 12.18 | -- | - | - | -- | -- |
| 400 | 1.34 | .004 | .005 | .025 | .05 | 6.58 | -- | - | - | -- | -- |
| 97 | .45 | .079 | .005 | .029 | .02 | -- | 6.00 | - | - | -- | -- |
| 257 | .49 | .079 | .005 | .029 | .02 | -- | -- | 2.35 | - | -- | -- |
| 209 | .50 | .079 | .005 | .029 | .02 | -- | -- | - | 8.48 | -- | -- |
| Commercial Steels | | | | | | | | | | | |
| 7 | .795 | .55 | -- | -- | .200 | -- | -- | - | - | -- | -- |
| S | 1.27 | .25 | -- | -- | .135 | -- | -- | - | - | -- | -- |
| 19 | 1.62 | .31 | -- | -- | .170 | -- | -- | - | - | -- | -- |
| 67 | 1.00 | 13.00 | -- | -- | -- | -- | -- | - | - | -- | -- |
| 69 | .60 | -- | -- | -- | -- | 4 | -- | 1 | 18 | -- | -- |
| Armor Plates | | | | | | | | | | | |
| E-7/8 | .165 | .44 | .013 | .018 | .235 | .14 | .305 | .105 | - | -- | 4.98 |
| N10-3 | .525 | .63 | .006 | .017 | .230 | 1.21 | .66 | .24 | - | -- | -- |
| P5-3 | .45/.55 | .40/.60 | .02 | .02 | .15/.25 | 1.10/1.30 | .60/.80 | .20/.30 | - | -- | -- |
| N4-3 | .545 | .57 | .011 | .017 | .24 | 1.29 | .60 | .24 | - | -- | -- |
| 1830-(D4) | .23 | .43 | .013 | .017 | .230 | .084 | .30 | .09 | - | .125 | 4.88 |
| 5A | .48 | .69 | -- | -- | .33 | 1.10 | .69 | .23 | - | .23 | .06 |

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Figure 1

a. Sample No. 234

Vacuum melt, Armco iron base, C 0.48%.

| | | |
|---|---|---|
| Nital Etch | X1000 | MA-1448 |

b. Same spot as in Figure 1a.

| | | |
|---|---|---|
| Murakami Etch approx. 20 min. | X1000 | MA-1449 |

c. Sample No. 437

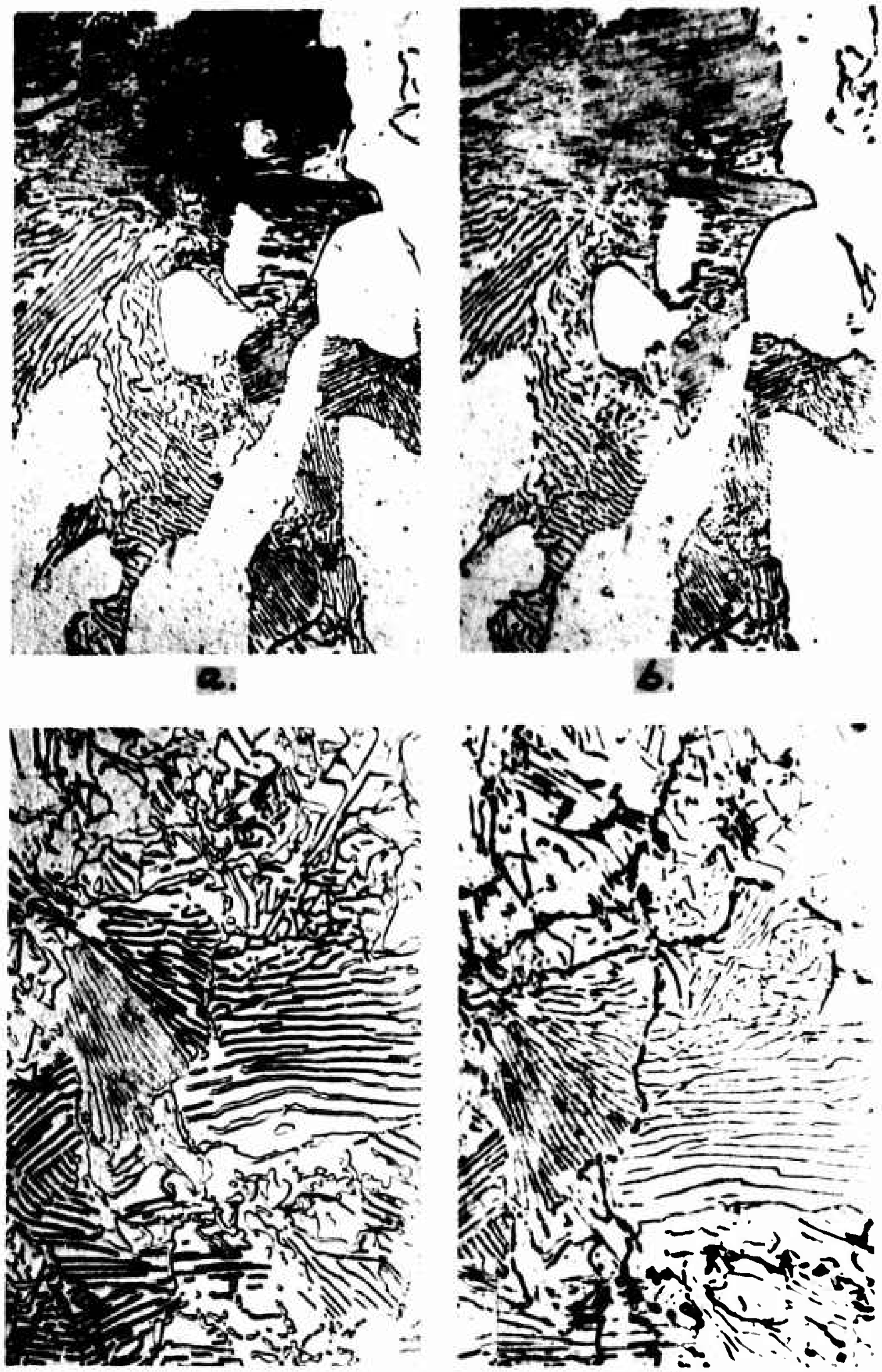
Vacuum melt, Armco iron base, C 1.06%.

| | | |
|---|---|---|
| Nital Etch | X1000 | MA-1450 |

d. Same spot as in Figure 1c.

| | | |
|---|---|---|
| Murakami Etch approx. 20 min. | X1000 | MA-1451 |

FIGURE 1.



a. b. c. d.

W.A.639-3845

Figure 2

a. Sample No. 600

Vacuum melt, Armco iron base, C 1.00%, P 0.26%.

Nital Etch X1000 MA-1452

b. Same spot as in Figure 2a.

Murakami Etch - 1 min. X1000 MA-1453

c. Sample No. 414

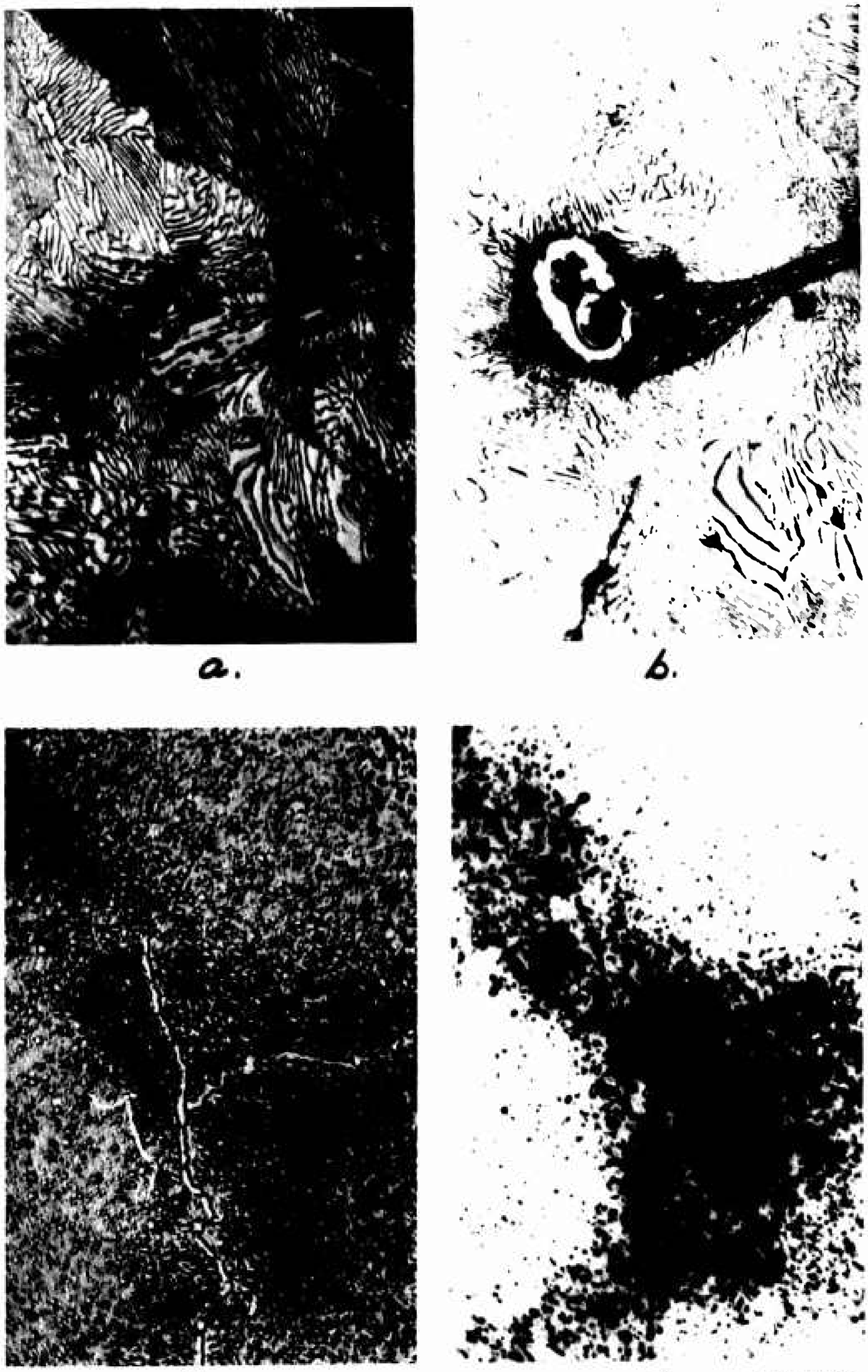
Vacuum melt, Armco iron base, C 0.52%, Cr 12.18%.

Nital Etch X1000 MA-1454

d. Same spot as in Figure 2c.

Murakami Etch - 30 sec. X1000 MA-1455

FIGURE 2.



a. b.

c. d. W.A.639-3846

Figure 3

a. Sample No. 400

Vacuum melt, Armco iron base, C 1.34%, Cr 6.58%.

Nital Etch X1000 MA-1456

b. Same spot as in Figure 3a.

Murakami Etch approx. 10 min. X1000 MA-1457

c. Sample No. 97

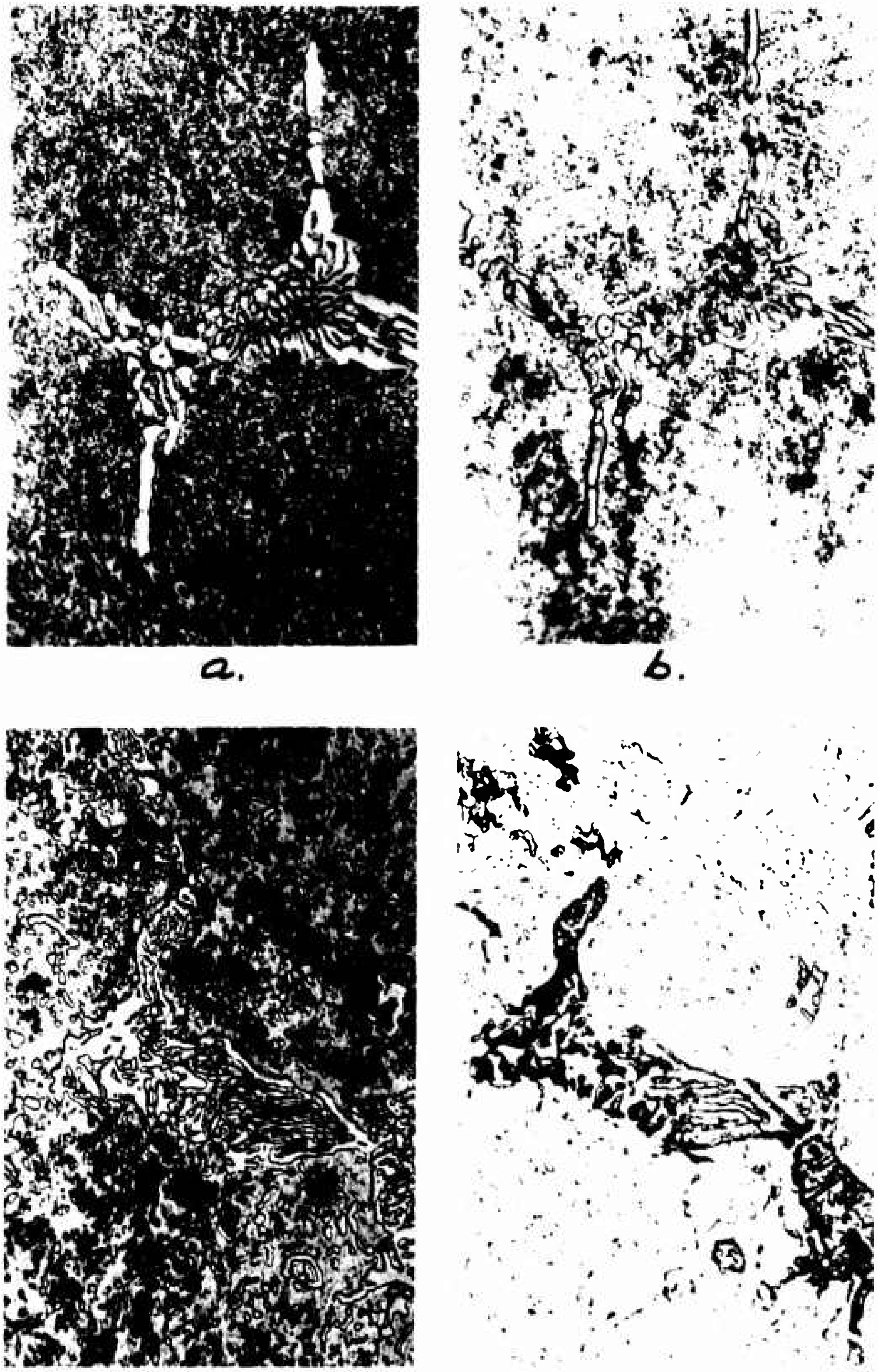
Vacuum melt, Armco iron base, C 0.45; Mo 6.00%.

Nital Etch X1000 MA-1458

d. Same spot as in Figure 3c.

Murakami Etch approx. 15 min. X1000 MA-1459

FIGURE 3.



a. b. c. d.

W.A. 639-3847

Figure 4

a. Sample No. 257

Vacuum melt, Armco iron base, C 0.49%, V 2.35%.

Nital Etch X1000 MA-1460

b. Same spot as in Figure 4a.

Murakami Etch approx. 15 min. X1000 MA-1461

c. Sample No. 209

Vacuum melt, Armco iron base, C 0.50, W 8.48%.

Nital Etch X1000 MA-1462

d. Same spot as in Figure 4c.

Murakami Etch approx. 15 min. X1000 MA-1463

FIGURE 4.

a.

b.

c.

d.

W.A.639-3848

Figure 5

a. Sample No. 7

Commercial hot rolled 1/4" rod, C 0.795%.

Nital Etch X1000 MA-1464

b. Same spot as in Figure 5b.

Murakami Etch - 30 min. X1000 MA-1465

FIGURE 5.

a.

b. W.A.639-3849

Figure 6

a. Sample No. S

Commercial spheroidized steel, C 1.27%.

Nital Etch X1000 MA-1466

b. Same spot as in Figure 6a.

Murakami Etch - 10 min. X1000 MA-1467

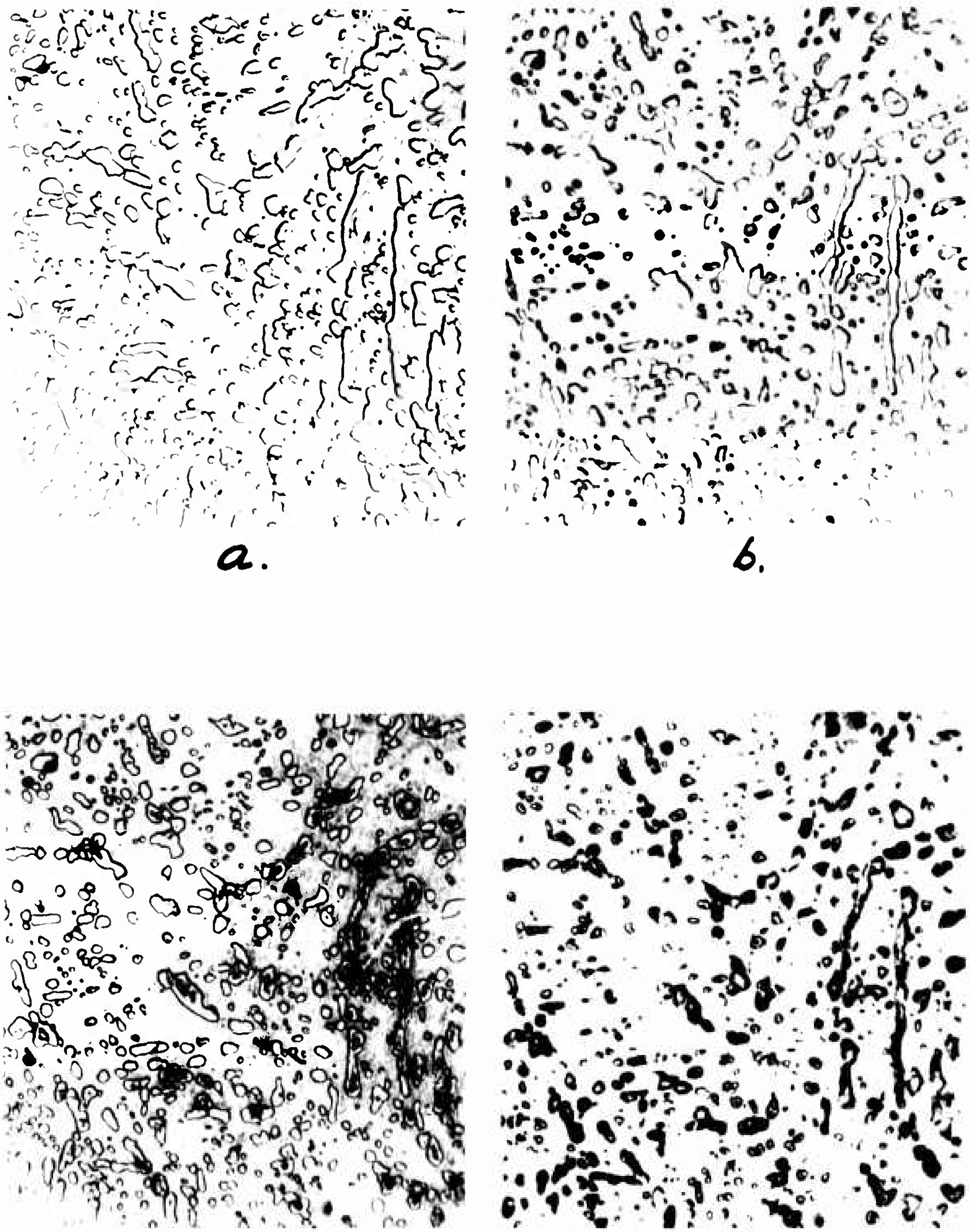
c. Same spot as in Figure 6a.

Murakami Etch - 30 min. X1000 MA-1468

d. Same spot as in Figure 6a.

Same etch as Figure 6c, but retouched on polishing wheel. X1000 MA-1469

FIGURE 6.



a. b.

c. d.

W.A.639-3850

## Figure 7

a. Sample No. 19

Commercial cast steel, C 1.62%.

Nital Etch X1000 MA-1470

b. Same spot as in Figure 7a.

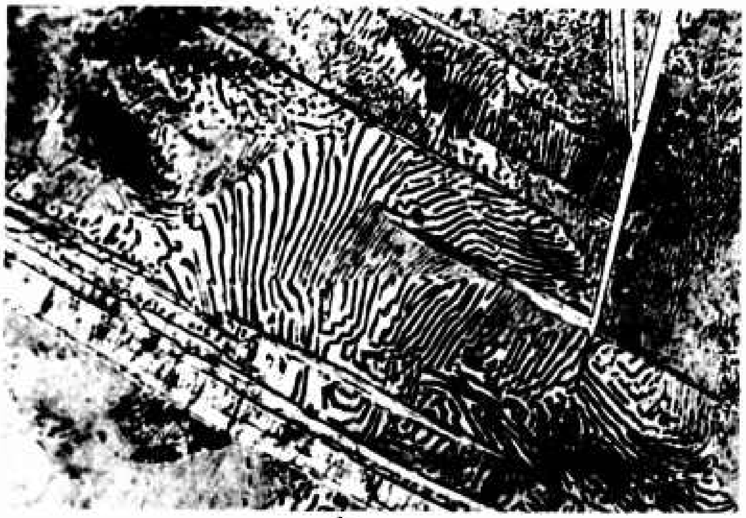
Murakami Etch - 30 min. X1000 MA-1471

c. Same spot as in Figure 7b.

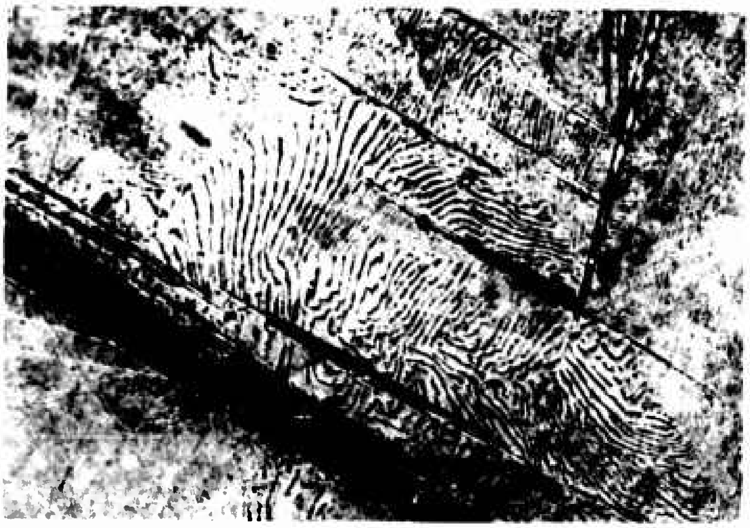
Murakami Etch - Retouched lightly on polishing wheel. X1000 MA-1472

FIGURE 7.

a.



b.



c.

W.A.030-3851

## Figure 8

a. Sample No. 67

Commercial cast Hadfield's manganese steel, C 1.00%, Mn 13.0%.

Nital Etch X1000 MA-1473

b. Same spot as in Figure 8a.

Murakami Etch - 15 min. X1000 MA-1474

c. Sample No. 69

Commercial cast high speed steel, C 0.60%, W 18.0%, Cr 4.0%, and V 1.0%.

Nital Etch X1000 MA-1475

d. Same spot as in Figure 8c. Note the troostitic area shown in Figure 8c is not revealed in this Figure due to the extremely short time etch. This might indicate that fine carbides require a longer etching time to be revealed.

Murakami Etch - 1+ sec. X1000 MA-1476

FIGURE 8.

a.

b.

c.

d.

W.A.639-385

Figure 9

a. 7/8" Nickel-Molybdenum face hardened armor plate No. E with a brittle case.
Pronounced chains of grain boundary carbides in the case.

Nital Etch X1000 MA-1480

b. Same spot as in Figure 9a. Note how Murakami's reagent makes the carbides stand out more prominently than does the nital etch.

Murakami's Etch - 10 min. X1000 MA-1482

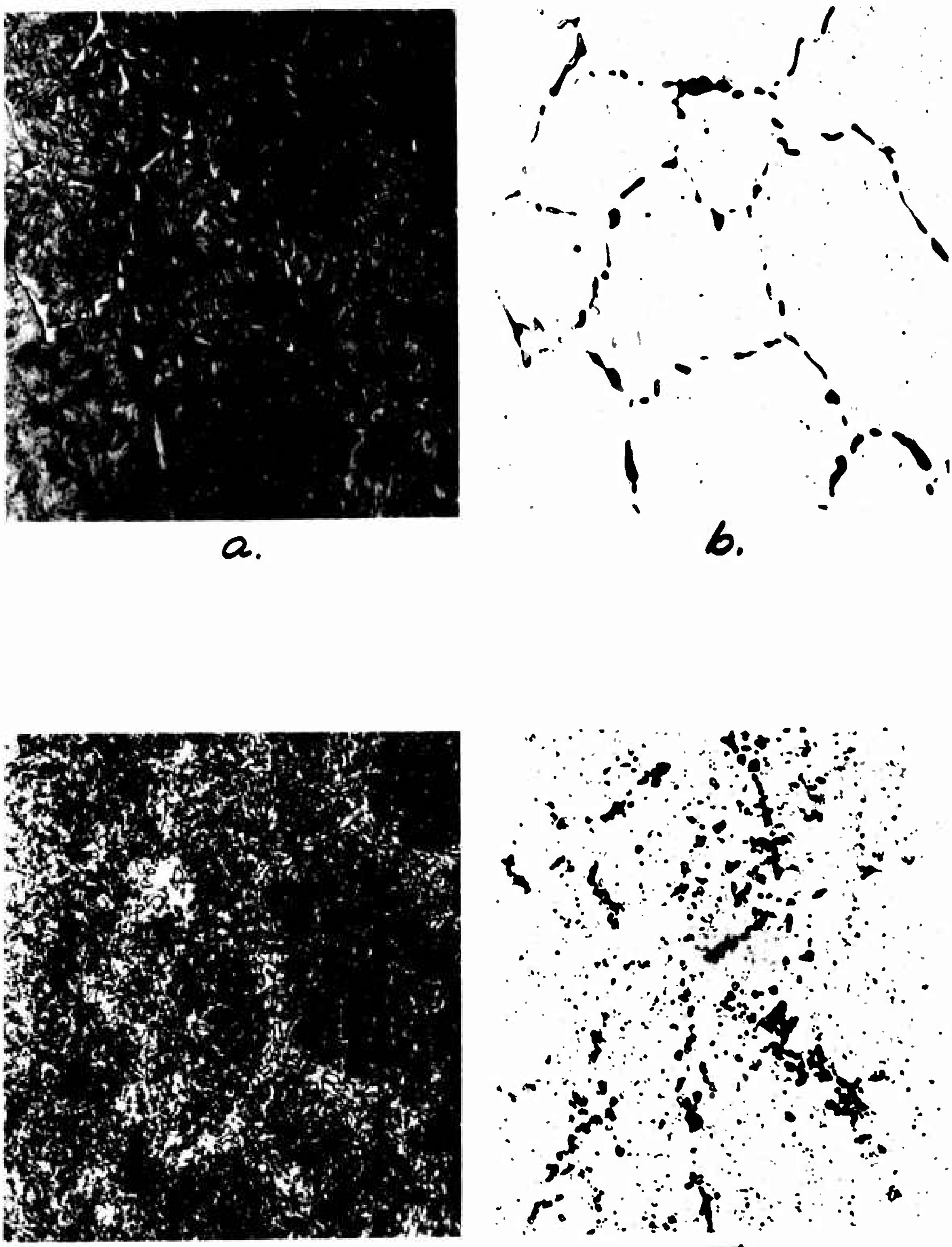
c. 1/2" Cr-Mo-V homogeneous armor plate No. N10-3. Ballistic limit 2350 f/s, Cal. .30 A.P. M1922. Buttoned and cracked. Carbides present in the grain boundaries.

Nital Etch X1000 MA-1772

d. Same spot as in Figure 9c. Carbides are more readily identified than with the nital etch.

Murakami Etch - 10 min. X1000 MA-1771

FIGURE 9.



a. b.

c. d. W.A.639-3853

Figure 10

a. 3/8" Cr-Mo-V homogeneous armor plate No. P5-3. Spalled. (Less brittle than plate No. N10-3. Figures 32 and 33.) Carbides difficult to identify.

Nital Etch X1000 MA-1778

b. Same spot as in Figure 10a. Carbides are more clearly revealed by the Murakami etch.

Murakami Etch - 10 min. X1000 MA-1777

c. 1/2" Cr-Mo-V homogeneous good quality armor plate No. N4-3.

Nital Etch X1000 MA-1774

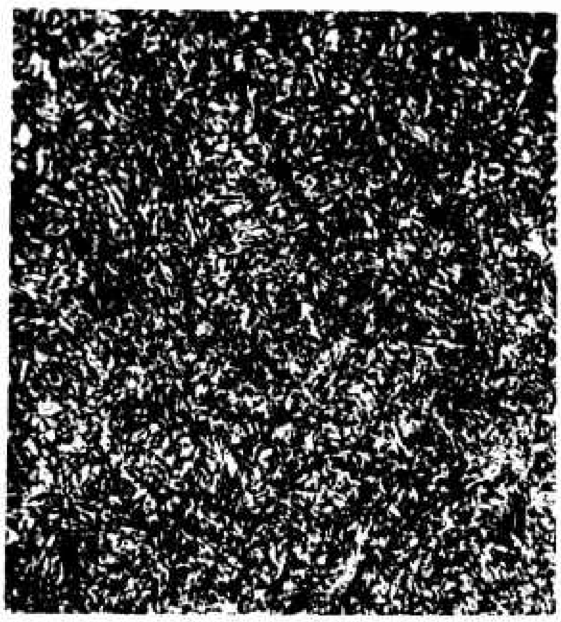
d. Same spot as in Figure 10c. Uniform distribution of fine carbides.

Murakami Etch - 10 min. X1000 MA-1773

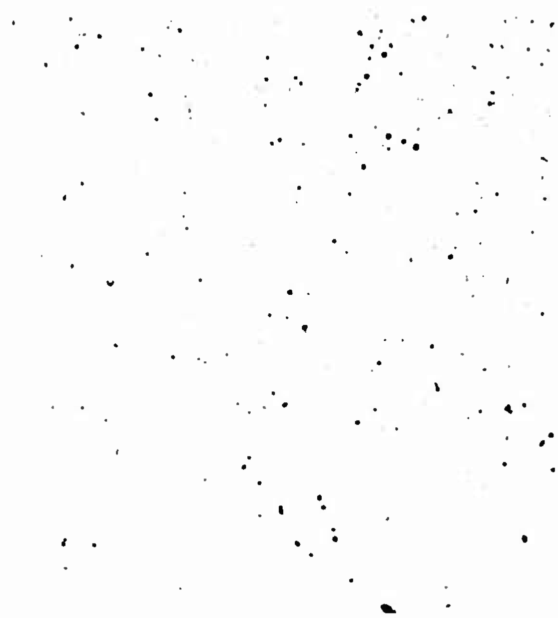
FIGURE 10.

a.

b.



c.



d.

W.A.639-3854

Figure 11

a. Plate No. 1830-(D4)

Good quality 1-1/2" homogeneous nickel-molybdenum armor plate.

Carbide condition as revealed by the Murakami etch, etching time 30 min. X1000 MA-3769

b. Same sample as in Figure 11a after etching in 10% chromic acid electrolytic etch, etching time 45 sec.

X1000 MA-3822

c. Same sample as in Figure 11b after etching 25 minutes in alkaline sodium picrate.

X1000 MA-3803

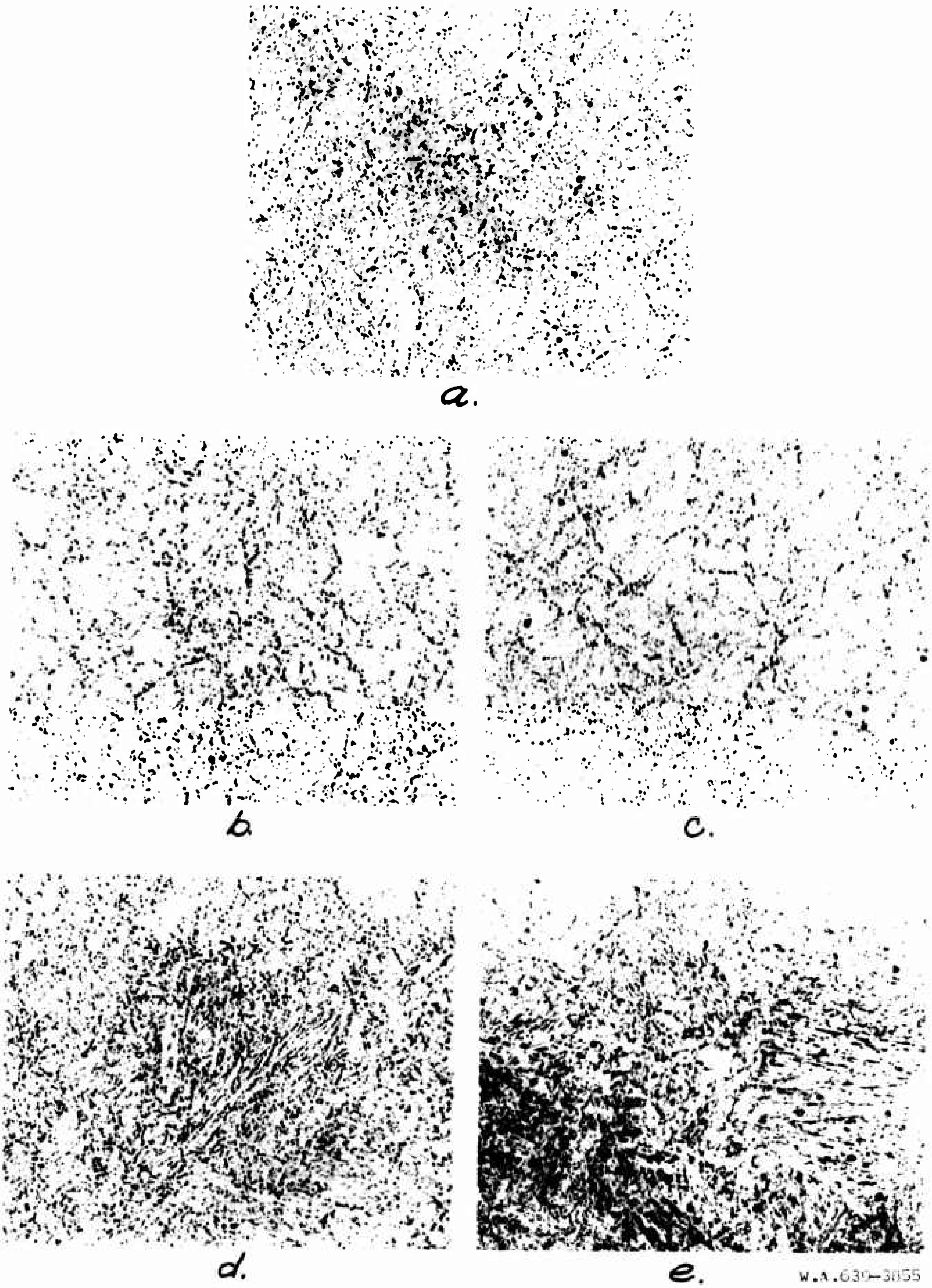
d. A relatively high carbon band in the same plate noted above in Figures 11a, 11b, and 11c after etching in 10% chromic acid electrolytic etch, etching time 45 sec.

X1000 MA-3821

e. Another area of the high carbon area shown in Figure 11c after etching 25 minutes in alkaline sodium picrate.

X1000 MA-3802

FIGURE 11.




W.A.639-3855

Figure 12

a. No. 5A

1/4" rolled Cr-Mo-V armor plate. Pronounced banding revealed by the sodium picrate etch. (Etch 25 minutes.) X100 MA-3790

b. Same plate as shown in Figure 12a after etching in 1% nital.

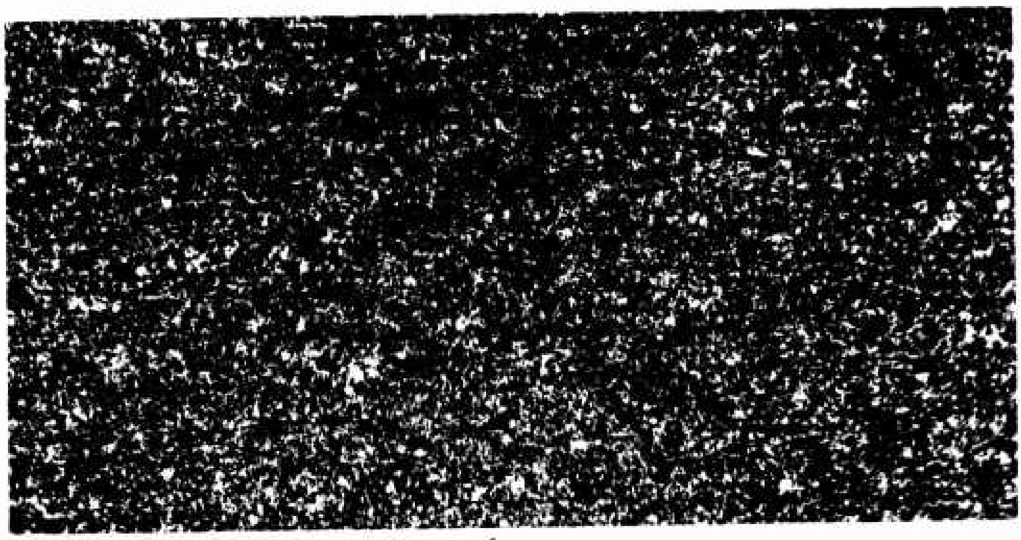
X100 MA-3794

c. Same plate as shown in Figure 12a after etching 1½ minutes in 10% chromic acid (electrolytic etch.)

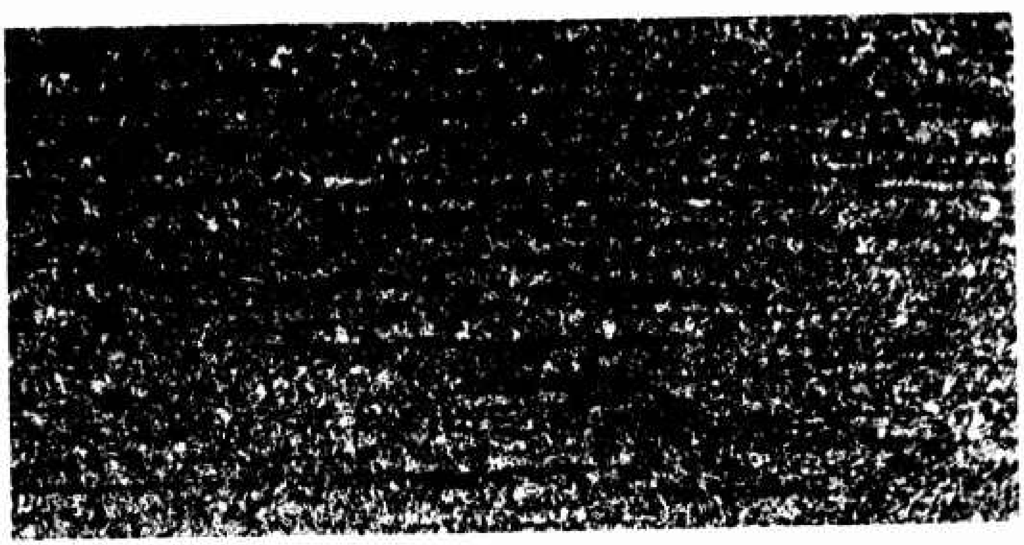
X100 MA-3799

FIGURE 12.

a.



b.



c.

W.A.63[illegible]

<u>Figure 13</u>

a. Rolled homogeneous Cr-Mo-V 1/4" plate No. 5A. Typical banding revealed by alkaline sodium picrate.

Etched in alkaline sodium picrate - 25 minutes.
X1000 MA-3784

b. Same plate as shown in Figure 13a showing microstructure after etching in 1% nital. Homogeneous structure with no banding revealed.

X1000 MA-3791

c. This photomicrograph illustrates a martensitic structure after etching in sodium picrate. Reproduced from Scientific Paper of the Bureau of Standards No. 452.

X1000

d. Showing same steel after tempering at 100°C for 20 days. Reproduced from Scientific Paper of the Bureau of Standards No. 452. "Structure of Martensitic Carbon Steels and Changes in Microstructure Which Occur upon Tempering" by H. S. Rawdon and S. Epstein.

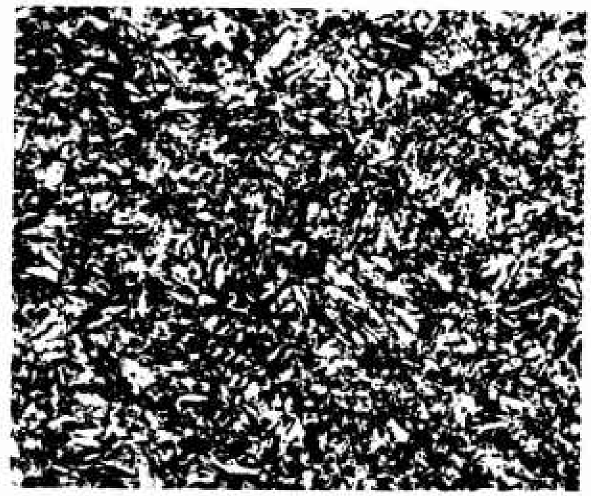
X1000

e. Cast manganese steel etched in boiling sodium picrate. Reproduced from A.I.M.M.E. Transactions, Vol. LXX, 1924, page 250. "Use of Sodium Picrate in Revealing Dendritic Segregation", by Albert Sauveur and V. N. Krivobok.

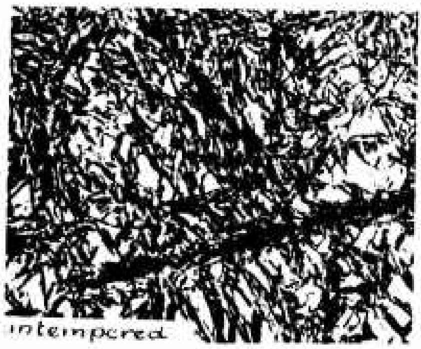
X50

FIGURE 13.

a.



b.




c.


d.

e.

W.A.637-3857